IS 11720-4 (1993): Methods of test for synthetic rubber, Part 4: Determination of volatile matter [PCD 13: Rubber and Rubber Products]

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IS 11720 (Part 4) : 1993

भारतीय मानक

(Reaffirmed 2003)

संश्लिष्ट रबड़ की परीक्षण पद्धतियों का मसौदा

भाग 4 वाष्पशील पदार्थ ज्ञात करना

Indian Standard

METHODS OF TEST FOR SYNTHETIC RUBBER

PART 4 DETERMINATION OF VOLATILE MATTER

UDC 678·7 : 543·813

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

May 1993

Price Group 1

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Rubber Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

The concerned committee has decided to prepare common methods of test for synthetic rubber under SR (Synthetic Rubber) series, namely, IS 11720 and this will be applicable to all types of synthetic rubbers being produced indigenously. This standard (Part 4) is the fourth in the series. The other standards of this series are as follows:

Part 1 Methods of test for synthetic rubber : Part 1 Determination of antioxidants (SR : 1);

Part 2 Methods of test for synthetic rubber : Part 2 Measurement of vulcanization characteristics with oscillating disc curemeter (SR : 2);

Part 3 Methods of test for synthetic rubber : Part 3 Determination of mooney viscosity; and

Part 5 Methods of test for synthetic rubber : Part 5 Determination of Ash.

In preparation of this standard, considerable assistance has been derived from ISO/DIS 248 'Rubber, raw — Determination of volatile matter content (Revision of ISO 248 : 1979)', issued by the International Organization for Standardization (ISO).

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS OF TEST FOR SYNTHETIC RUBBER

PART 4 DETERMINATION OF VOLATILE MATTER

1 SCOPE

1.1 This standard (Part 4) prescribes two methods, a hot mill method and an oven method, for the determination of moisture and other volatile matter content in raw synthetic rubber.

1.2 These methods are suitable for the determination of the volatile matter content in rubbers having an unsaturated carbon chain, that is, rubbers derived at least partly from diolefins for example, acrylate butadiene rubber (ABR); Butadiene rubber (BR), chloroprene rubber, isobutene isoprene rubber (butyl rubber IIR), isoprene rubber, synthetic (IR), acrylonitrile butadiene rubber (nitrile rubber), styrene butadiene rubber (SBR) etc. They may also be used for other rubbers, but in these cases it is necessary to prove that the change in mass is solely due to loss of original volatile matter and not due to rubber degradation.

1.3 The hot mill method is not applicable to synthetic isoprene rubbers or to rubbers too difficult to handle on a hot-mill or to rubbers in powdered or chip form.

1.4 The two test methods do not necessarily give identical results. Therefore, in case of dispute the oven method is the reference method.

2 REFERENCES

2.1 The Indian Standards listed below are necessary adjuncts to this standard:

IS No.	*Title*
5599 : 1970	Rubber, raw natural and synthetic — Methods of sampling and sample preparation (*first revision*)
3660 (Part 51) : 1972	Methods of test for natural rubber : Determination of ash, total copper, manganese, rubber hydrocarbon, viscosity (shearing disk viscometer), and mixing and vulcanizing of rubber in standard compound (*first revision*)

3 OUTLINE OF THE METHOD

3.1 Hot Mill Method

Test portion is sheeted out on a heated mill until all volatile matter is driven off. Loss in mass during milling is calculated and expressed as volatile matter content.

3.2 Oven Method

Test portion is homogenized using a laboratory mill and sheeted out. Dry in an oven to constant mass. Calculate the volatile matter content as the mass lost during this procedure, together with the mass lost during any homogenization of the piece.

4 HOT MILL METHOD

4.1 Apparatus

4.1.1 *Mixing Mill*

4.1.2 *Analytical Balance* — Accurate to 0·1 g.

4.2 Procedure

4.2.1 Sheet out a test piece of about 250 g in accordance with IS 5599 : 1970. Weigh to the nearest 0·1 g before and after homogenization (masses M_1 and M_2 respectively).

4.2.2 Adjust the clearance of the mill rolls to 0·25 mm $\pm$ 0·05 mm, using lead strips as specified in IS 5599 : 1970. Maintain the surface temperature of the rolls at 100 $\pm$ 5°C.

4.2.3 Pass a weighed test portion (mass M_3) repeatedly through the mill for 4 minutes. Do not allow the test portion to band and take care to prevent any loss of rubber. Weigh the test portion to the nearest 0·1 g. Pass the test portion through the mill for an additional 2 minutes and reweigh. If the masses at the end of the 4 minutes and 6 minutes periods differ by less than 0·1 g, calculate the volatile matter content; if not, continue passing the test portion through the mill for 2 minutes period until the mass does not decrease by more than 0·1 g between successive weighings (final mass M_4). Before each weighing, allow the rubber to cool to room temperature in a desiccator.

4.2.4 If the rubber is flaky or becomes sticky in the mill roll, making weighing difficult or impossible, the oven method shall be used.

4.3 Expression of Results

The volatile matter content is given, as a percentage by mass, by the formula:

$$\text{Volatile matter, percent by mass} = \left(1 - \frac{M_2 \times M_4}{M_1 \times M_3} \right) \times 100$$

where

M_1 = mass in grams, of the test portion before homogenization;

M_2 = mass in grams, of the test portion after homogenization;

M_3 = mass in grams, of the test portion before milling; and

M_4 = mass, in grams, of the test portion after milling.

5 OVEN METHOD

5.1 Apparatus

5.1.1 *Oven*

Ventilated, preferably air-circulating type, capable of being controlled at 105°C ± 5°C.

5.1.2 *Analytical Balance* — Accurate to 0·1 g.

5.2 Procedure

5.2.1 Select a piece of about 250 g and homogenize in accordance with the procedure specified in IS 5599 : 1970. Weigh the piece to the nearest 0·1 g before and after this homogenization (masses M_5 and M_6 respectively).

5.2.2 With the mill set at 70 ± 5°C and with a mill opening which will produce a sheet of less than 2 mm thickness, pass a test portion of 10 g taken from the homogenized piece and weighed to a nearest of 0·1 mg (mass M_7), twice between the rolls.

5.2.3 If this sheeting is impossible, take a 10 g test portion from the homogenized piece and cut it by hand into small cubes with edges of approximately 2 mm. Place the cubes on a watch-glass or an aluminium tray to facilitate weighing. Weigh to the nearest 0·1 mg (mass M_7).

5.2.4 Alternatively, if the rubber is in powdered or chip form select a test portion of about 10 g taken at random and place it on a watch-glass or an aluminium tray to facilitate weighing. Weigh to the nearest 0·1 mg (mass M_7).

5.2.5 Place the test portion for 1 h in the oven (**5.1**), controlled at 105 ± 5°C with the ventilators open and with the circulating fan, if fitted, switched on. Arrange the rubber so as to present the largest possible surface area to the hot air. Allow to cool in a desiccator and weigh. Repeat the heating for further 30 minutes period until the mass does not decrease by more than 1 mg between successive weighings (final mass M_8).

5.3 Expression of Results

5.3.1 If the test portion was taken from a homogenized piece (*see* **5.2.2**), the volatile matter content is given, as a percentage by mass, by the formula:

$$\text{Volatile matter content, percent by mass} = \left(1 - \frac{M_6 \times M_8}{M_5 \times M_7} \right) \times 100$$

where

M_5 = mass, in grams, of the piece before homogenization;

M_6 = mass, in grams, of the piece after homogenization;

M_7 = mass, in grams, of the test portion as taken from the piece;

M_8 = mass, in grams, of the test portion after oven drying.

5.3.2 If the test portion was taken directly from the powdered or chip form lot (*see* **5.2.4**), the volatile matter content is given, as a percentage by mass, by the formula:

$$\text{Volatile matter content, percent by mass} = \left(\frac{M_7 \times M_8}{M_7} \right) \times 100$$

where

M_7 and M_8 are as defined in **5.3.1**.

6 TEST REPORT

6.1 The test report shall include the following particulars:

a) Reference to this Indian Standard;

b) All details necessary for the full identification to the piece;

c) The method used (hot-mill or oven);

d) Whether the 10 g test portion were taken from a homogenized piece (*see* **5.2.2**) or directly from the powdered or chip from (*see* **5.2.4**);

e) The result obtained on each test portion;

f) Any unusual features noted during the determination;

g) Any operation not included in this Indian Standard or regarded as optional; and

h) The date of test.

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BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones : 331 01 31, 331 13 75

Telegrams : Manaksanstha
(Common to all offices)

Regional Offices :	Telephone
Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002	331 01 31 331 13 75
Eastern : 1/14 C. I. T. Scheme VII M, V. I. P. Road, Maniktola, CALCUTTA 700054	37 84 99, 37 85 61 37 86 26, 37 86 62
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